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परिवहनी गैस सिलिंडर — गैस की संघटक से सिलिंडर एवं वाल्व सामग्रियों की अनुरूपता

भाग 4 हाइड्रोजन से विखंडनीयता की प्रतिरोधी धात्विक सामग्री के चयन की परीक्षण पद्धतियाँ

Indian Standard

TRANSPORTABLE GAS CYLINDERS — COMPATIBILITY OF CYLINDER AND VALVE MATERIALS WITH GAS CONTENTS

PART 4 TEST METHODS FOR SELECTING METALLIC MATERIALS RESISTANT TO HYDROGEN EMBRITTLEMENT

ICS 23.020.30

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 2013 **Price Group 7**

Gas Cylinders Sectional Committee, MED 16

NATIONAL FOREWORD

This Indian Standard (Part 4) which is identical with ISO 11114-4 : 2005 'Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents — Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Gas Cylinders Sectional Committee and approval of the Mechanical Engineering Division Council.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

> Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respective places are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 9809-1 : 1999[1] Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 1: Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa	IS 7285 (Part 2) : 2004 Refillable seamless steel gas cylinders — Specification: Part 2 Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa (112 kgf/mm^2)	Technically Equivalent
ISO 9809-3 : 1999[1] Gas cylinders – Refillable seamless steel gas cylinders — Design, construction and testing — Part 3: Normalized steel cylinders	IS 7285 (Part 1): 2004 Refillable seamless steel gas cylinders — Specification: Part 1 Normalized steel cylinders	do

The technical committee has reviewed the provisions of the following International Standards referred in this adopted standard and has decided that they are acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 4287 : 1997	Geometrical product specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters
ISO 7539-1 : 1987	Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures
ISO 7539-6 : 2003	Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement
ISO 9809-2 : 2010	Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 2: Quenched and tempered steel cylinders with tensile strength greater than or equal to 1 100 MPa
ISO 11114-1 : 1997	Transportable gas cylinders — Compatibility of cylinder and valve material with gas contents — Part 1: Metallic materials

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[1] Since revised in 2010.

(*Continued on third cover*)

Indian Standard

TRANSPORTABLE GAS CYLINDERS — COMPATIBILITY OF CYLINDER AND VALVE MATERIALS WITH GAS CONTENTS

PART 4 TEST METHODS FOR SELECTING METALLIC MATERIALS RESISTANT TO HYDROGEN EMBRITTLEMENT

1 Scope

This part of ISO 11114 specifies test methods and the evaluation of results from these tests in order to qualify steels suitable for use in the manufacture of gas cylinders (up to 3 000 l) for hydrogen and other embrittling gases.

This part of ISO 11114 only applies to seamless steel gas cylinders.

The requirements of this part of ISO 11114 are not applicable if at least one of the following conditions for the intended gas service is fulfilled [1]:

- the working pressure of the filled embrittling gas is less than 20 % of the test pressure of the cylinder;
- the partial pressure of the filled embrittling gas of a gas mixture is less than 5 MPa (50 bar) in the case of hydrogen and other embrittling gases, with the exception of hydrogen sulphide and methyl mercaptan in which cases the partial pressure shall not exceed 0,25 MPa (2,5 bar).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4287, *Geometrical product specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 7539-1:1987, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

ISO 7539-6:2003, *Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*

ISO 9809-1, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 1: Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa*

ISO 9809-2, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 2: Quenched and tempered steel cylinders with tensile strength greater than or equal to 1 100 MPa*

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1) In such cases the cylinders may be designed as for ordinary (non embrittling) gases.

ISO 9809-3, *Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 3: Normalized steel cylinders*

ISO 11114-1:1997, *Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents — Part 1: Metallic materials*

ISO 11120, *Gas cylinders — Refillable seamless steel tubes for compressed gas transport, of water capacity between 150 l and 3 000 l — Design, construction and testing*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply. Some of the definitions used are based upon those in ISO 7539-1:1987.

3.1.1
embrittling gases
gases listed in ISO 11114-1:1997, Table A.3, group 2 and Table A.12, group 11

3.1.2
hydrogen rupture pressure, P_{H2}
maximum pressure recorded during the hydrogen rupture pressure test

3.1.3
helium rupture pressure, P_{He}
maximum pressure recorded during the helium rupture pressure test

3.1.4
hydrogen embrittlement index
maximum value of the ratio $P_{\mathrm{He}}/P_{\mathrm{H2}}$ as a function of the pressure rise rate

3.1.5
environmentally-assisted cracking
synergistic effect on a metal caused by the simultaneous action of a particular environment and a nominally static tensile stress, which results in the formation of cracking

3.1.6
threshold stress
stress above which a crack will initiate and grow, for the specified test conditions

3.1.7
plane strain stress intensity factor, K_1
function of applied load, crack length and specimen geometry having dimensions of stress × length

NOTE K_1 uniquely defines the elastic stress field intensification at the tip of a crack subjected to opening mode displacements.

3.1.8
threshold stress intensity factor for susceptibility to environmentally-assisted cracking, $K_{1\mathrm{H}}$
stress intensity factor above which an environmentally-assisted crack will initiate and grow, for the specified test conditions under conditions of high constraint to plastic deformation, i.e. under essentially plane strain conditions

3.1.9
HAC
hydrogen assisted cracking

3.2 Symbols

For the purposes of this document, the following symbols apply.

a	effective crack length measured from the crack tip to the loading plane
a_0	average value of a
B	specimen thickness
e_m	the mean disc thickness
E	modulus of elasticity
K_IAPP	applied elastic stress-intensity
K_1H	threshold stress intensity factor
m	elastic displacement per unit load
P	applied load
P_r	actual rupture pressure
P_r'	corrected rupture pressure
$P_\mathrm{r}'{}_\mathrm{H2}$	corrected hydrogen rupture pressure
$P_\mathrm{r}'{}_\mathrm{He}$	theoretical helium rupture pressure corresponding to the same pressure rise rate as for the hydrogen test, calculated by regression from the corrected helium rupture pressure
R_m	actual value of tensile strength
$R_\mathrm{P0,2}$	average of measured yield stress of three specimens from the test cylinder representing the HAC test specimen's location at room temperature
V	crack-mouth opening displacement (CMOD) defined as the mode 1 (also called opening-mode) component of crack displacement due to elastic and plastic deformation, measured at the location on a crack surface that has the greatest elastic displacement per unit load, m
W	effective width of a compact specimen, measured from the back face to the loading plane
Y	stress intensity factor coefficient derived from the stress analysis for a particular specimen geometry, which relates the stress intensity factor for a given crack length to the load and specimen dimensions

4 General requirements

The test methods as described in Clause 5 are valid for working pressure up to 300 bar. For higher working pressures additional verification shall be undertaken.

The tests shall be performed for selecting steels for hydrogen cylinders. Chromium–molybdenum steels, quenched and tempered with a guaranteed maximum actual ultimate tensile strength of 950 MPa, do not need to be tested and can safely be used for the construction of hydrogen cylinders. For carbon–manganese steels, different limits may apply (see ISO 9809-1).

The tests described in Clause 5 are “qualification tests” for a given steel. This means that the tests need not be repeated for each “type” of cylinder once a steel has been qualified for a specific design strength level.

The test samples shall be taken from a representative cylinder or from a piece of tube (for long cylinders according to ISO 11120), representative of the relevant manufacturing process including heat treatment.

The test samples shall have a mechanical strength not lower than the maximum intended tensile strength to be used for the cylinders to be manufactured. If it is intended later to increase the maximum strength of the steel, a new qualification test shall be performed.

With respect to the possible variation of the chemical composition, the chemistry of the steel tested shall be recorded in the qualification test report and the difference in chemistry for the steels actually used for the cylinders shall not exceed the "permissible difference" according to ISO 9809-2. In addition, for sulphur and phosphorus, these permissible differences are limited to 0,005 % and 0,010 %, respectively. In no case shall the phosphorus content of either the qualification or the production cylinders exceed 0,015 %.

With respect to the heat treatment, the manufacturer shall specify the relevant temperatures and times, and the quenching conditions (if relevant). Any modification to the heat treatment needing a new type approval according to ISO 9809-2 requires a new qualification test.

For the qualification of a given steel for the manufacturing of gas cylinders, method A, B, or C can be used (see 5.1, 5.2 and 5.3, respectively). Additionally, tensile tests shall be carried out (see 5.4).

5 Test methods

5.1 Disc test (method A)

5.1.1 Principle of test

A mounted test piece in the shape of a disc is subjected to an increasing gas pressure at constant rate to burst or to crack. The embrittling effect of hydrogen is evidenced by comparing the hydrogen rupture pressures P_{H2} with the helium rupture pressures P_{He}, helium being chosen as a reference gas.

The ratio $P_{\text{He}}/P_{\text{H2}}$ shall be determined.

The lower this ratio, the better will the steel type behave in the presence of hydrogen. This ratio is dependent on the pressure rise rate, which shall remain constant during the whole test.

NOTE 1 Hydrogen rupture pressures also depend on the hydrogen purity. Oxygen or traces of water vapour can partially inhibit the hydrogen embrittlement effect.

NOTE 2 The test can be carried out with any other embrittling gas or gas mixture (e.g. H_2S, hydrides). The embrittlement index of the considered gas will then be defined similarly.

5.1.2 Test conditions and procedure

5.1.2.1 Sample disc

The sample disc shall be flat and ground (or machined to an equivalent surface finish), and shall have the following characteristics.

Dimensions:

- Diameter: $58\,^{0}_{-0,5}$ mm.
- Thickness: 0,75 mm ± 0,005 mm.
- Flatness: less than 1/10 mm deflection.

Surface condition (both sides):

- Roughness: Ra value (see ISO 4287) less than 0,001 mm. The roughness of the samples used for both H_2 and He tests shall be equivalent.
- No trace of oxides.

The following operations shall be performed to verify the sample quality:

- Immediately after the final preparation and prior to the testing, store the samples in a dry atmosphere, e.g. a desiccator.
- Degrease the sample and check thickness at 4 points taken 90° apart to define a mean thickness.
- Determine the disc's hardness (e.g. Vickers) over its outer circumference to verify that machining has not altered the original material properties.

5.1.2.2 Cell and other apparatus

The cell (see Figure 1) is composed of 2 stainless steel flanges embedding the disc. A volume of approximately 5 cm^3 is provided below the disc. Above the disc a high-strength steel ring is mounted ($R_m \geqslant$ 1 100 MPa). The internal diameter is 25,5 mm and the ring curvature radius at the mounting diameter level is 0,5 mm.

Gas discharges can occur after disc bursting either through the lower flange or upper flange discharge port to the atmosphere. This permits the evacuation of the installation and a check of the hydrogen purity and freedom from either oxygen ($O_2 <$ 1 µl/l) or water vapour ($H_2O <$ 3 µl/l) [2]. It also permits regulation of the gas flow and adjustment of the pressure rise rate.

The sealing device shall be an elastomer O-ring for helium testing, and for hydrogen testing at rates of more than 10 bar/min. For hydrogen testing at rates not exceeding 10 bar/min, indium O-rings shall be used.

For the flange assembly, ten high-strength steel bolts should be used, size typically M10 or equivalent. The tightening torque shall be 30 N·m for elastomer O-rings and 100 N·m for indium O-rings.

Hydrogen and helium shall be stored in high-pressure containers connected to the test cells. A flow control valve between the high-pressure container and the cell shall be used to adjust the pressure rise rate.

5.1.2.3 Test procedure

For a satisfactory outcome of the test performances, the following operations shall be carried out.

- Evacuation of the cell by pumping to eliminate any traces of air or moisture absorbed by the walls. Purging with the gas to be used, followed by vacuum pumping, can also be used to improve the cleaning efficiency.
- Check of the gas purity before testing.
- Adjustment of the gas flow to achieve the appropriate pressure rise rate.
- Isolation of the cell (at the start of pressure rise).

The rate of pressure rise shall be monitored for the duration of the entire test. It shall be regular and kept as constant as possible, neglecting the gas compressibility factor with pressure.

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[2] 1 µl/ = 1 ppm. The use of ppm is deprecated.

The rupture pressures shall be noted from the recorded readings at the end of the test. The pressure gauge shall be accurate to ± 2 % for the intended measured rupture pressure.

The tests shall be carried out with hydrogen (for quality, see 5.2.2.3) and with helium ($H_2O < 3$ µl/l) for a range of pressure rise rates evenly distributed between 0,1 bar/min and 1 000 bar/min. When using these latter pressure rates, the minimum value for the rupture pressure shall be established (see Figure 2). This minimum value shall be used to run the rest of the tests. It is generally considered that 6 helium tests and 9 hydrogen tests (i.e 15 tests in total) are enough for a thorough material evaluation.

5.1.3 Treatment and interpretation of test results

5.1.3.1 Disc thickness

Rupture pressure P_r shall be systematically corrected for deviation from the "ideal" value corresponding to the test of a standard thickness disc.

The corrected rupture pressure P_r', shall be given by the formula:

$$P_\mathrm{r}' = \frac{P_\mathrm{r} \times 0{,}75}{e_\mathrm{m}}$$

where e_m is the mean disc thickness.

5.1.3.2 Presentation and interpretation of test results

Rupture pressures, corrected as indicated in 5.1.3.1, shall be plotted against the mean pressure rise rate (actual rupture pressure divided by the test duration) expressed in bar/min (see Figure 2).

For each hydrogen test, calculate the ratio

$$P_\mathrm{r}'{}_\mathrm{He}/P_\mathrm{r}'{}_\mathrm{H2}$$

where

$P_\mathrm{r}'{}_\mathrm{He}$ is the theoretical helium rupture pressure corresponding to the same pressure rise rate as for the hydrogen test, calculated by regression from the corrected helium rupture pressure;

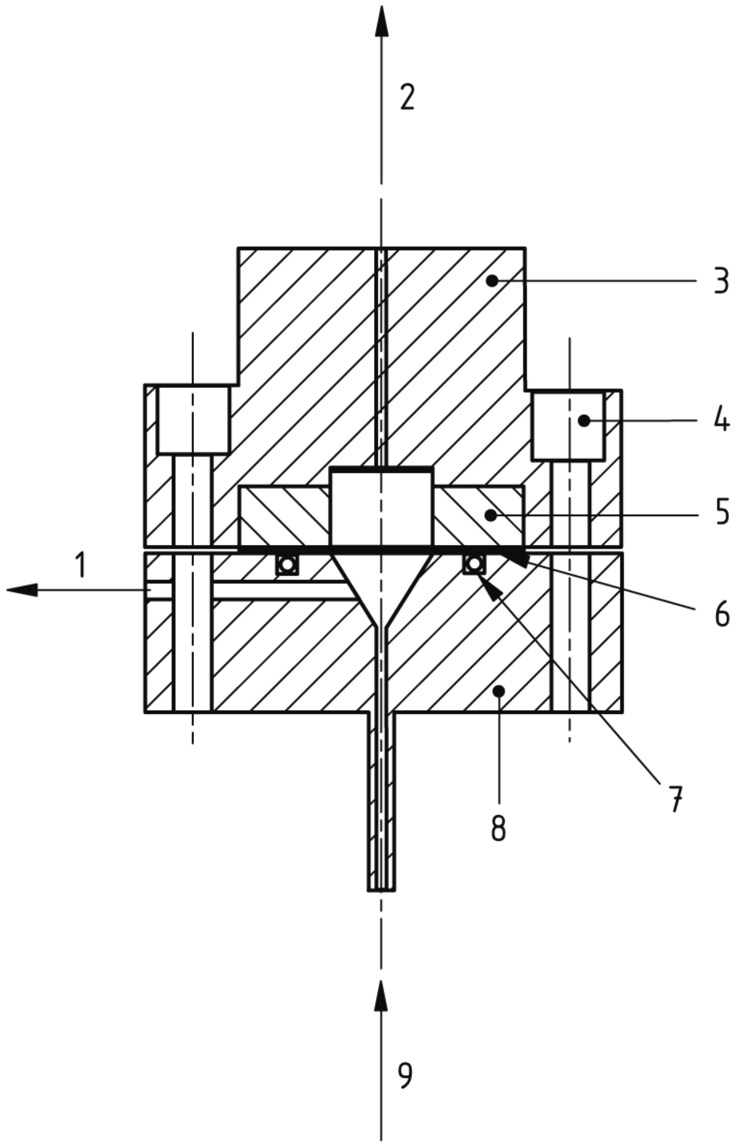
$P_\mathrm{r}'{}_\mathrm{H2}$ is the corrected hydrogen rupture pressure.

Ratios of $P_\mathrm{r}'{}_\mathrm{He}/P_\mathrm{r}'{}_\mathrm{H2}$ shall be plotted against the pressure rise rate (see Figure 3).

Test results shall be interpreted bearing in mind that the embrittlement index of a material is the maximum value of the above-mentioned ratio and that the material shall be considered as suitable for compressed hydrogen cylinders if the index is less than or equal to 2.

5.1.4 Test report

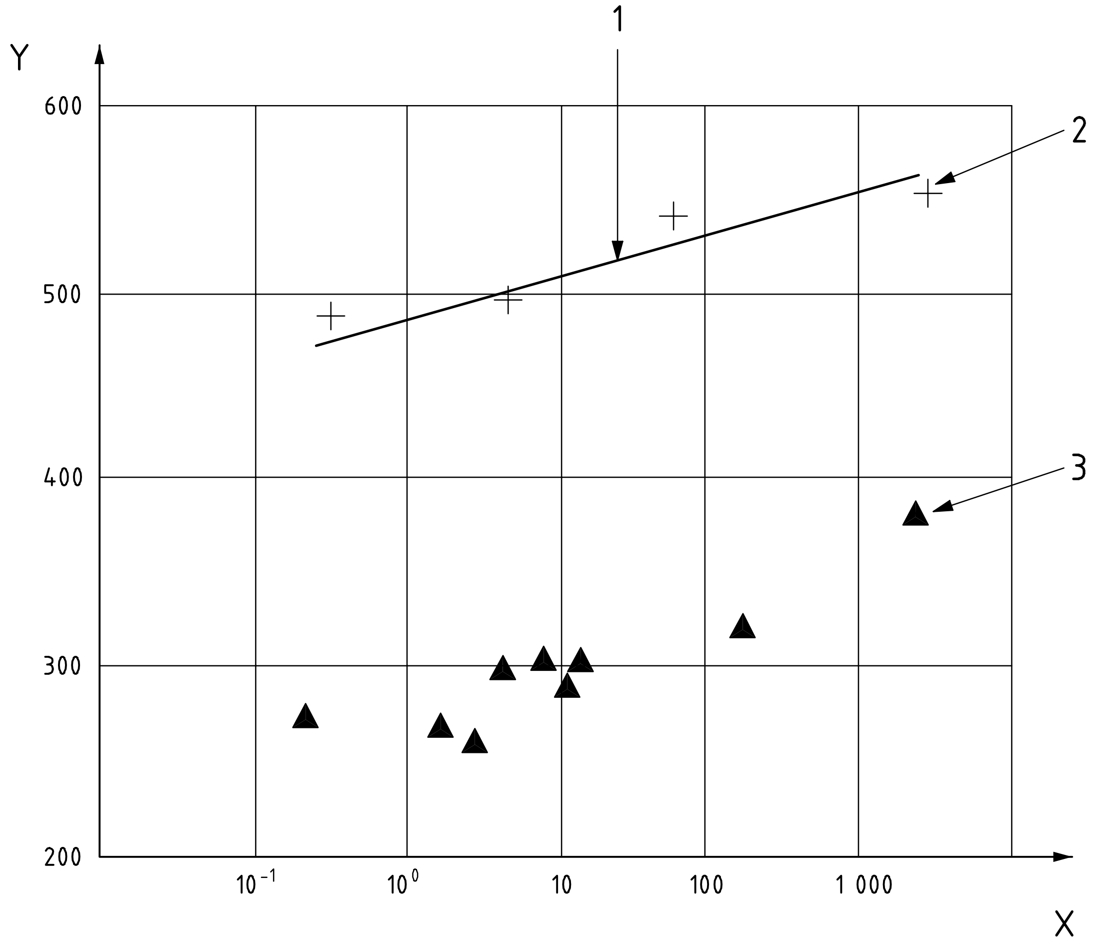
The detailed test conditions (gas purity, rupture pressure, hardness of the specimen, ratio $P_\mathrm{r}'{}_\mathrm{He}/P_\mathrm{r}'{}_\mathrm{H2}$, embrittlement index) and the cylinder design drawing including the material properties, chemical composition and heat treatment conditions of the tested cylinder shall be reported.



Key

1 port for evacuation and flow adjustment
2 discharge port
3 upper flange
4 bolt hole
5 high-strength steel ring
6 disc
7 O-ring
8 lower flange
9 gas inlet

Figure 1 — Test installation (test cell)



X pressure rise rate (bar/min)

Y corrected rupture pressure (bar)

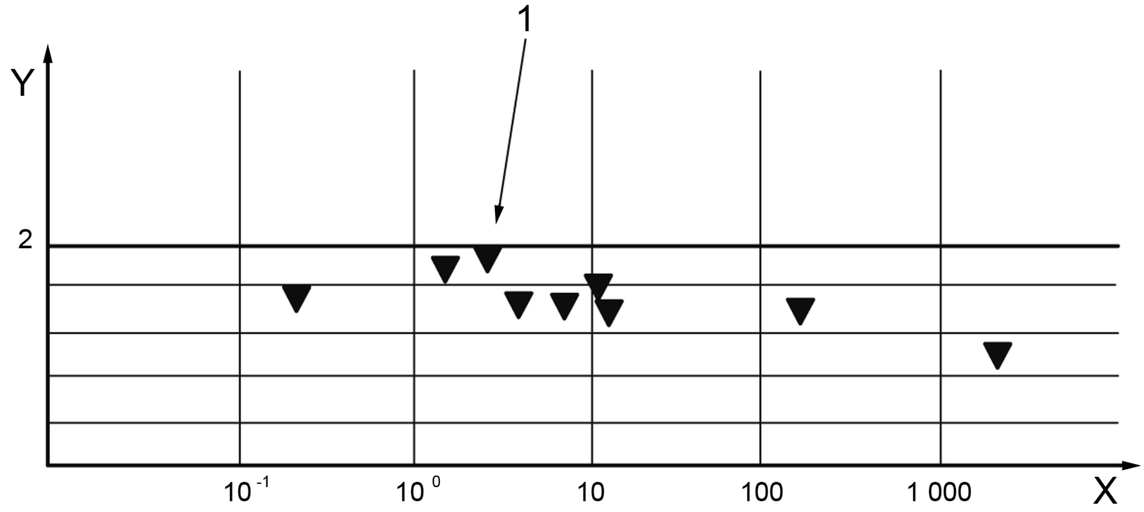
Key

1 regression curve

2 helium corrected rupture pressures ($P_r'_{He}$)

3 hydrogen corrected rupture pressures ($P_r'_{H2}$)

Figure 2 —Examples of hydrogen and helium corrected rupture pressures as a function of the pressure rise rate



X pressure rise rate (bar/min)

Y $P_{\mathrm{r}}'{}_{\mathrm{He}}/P_{\mathrm{r}}'{}_{\mathrm{H2}}$

Key

1 hydrogen embrittlement index

Figure 3 — Examples of the ratio $P_{\mathrm{r}}'{}_{\mathrm{He}}/P_{\mathrm{r}}'{}_{\mathrm{H2}}$ as a function of the pressure rise rate

5.2 Fracture mechanic test (method B)

5.2.1 Principle of the test method

This specifies a method for the determination of the threshold stress intensity factor ($K_{1\mathrm{H}}$) for susceptibility to cracking of metallic materials in gaseous hydrogen.

The procedure covers the use of machined compact tension test pieces, as described in ISO 7539-6, for the determination of the threshold stress intensity factor, as described in ISO 7539-1.

The test involves a specimen containing a machined notch. This notch is extended by fatigue cracking to an incrementally increasing tensile load during exposure to a pressurised, gaseous hydrogen environment. The objective is to quantify the conditions under which environmentally-assisted crack propagation can occur.

If the specimens pass the test requirements, then the material is characterized as suitable for gas cylinders filled with compressed hydrogen.

5.2.2 Test procedure

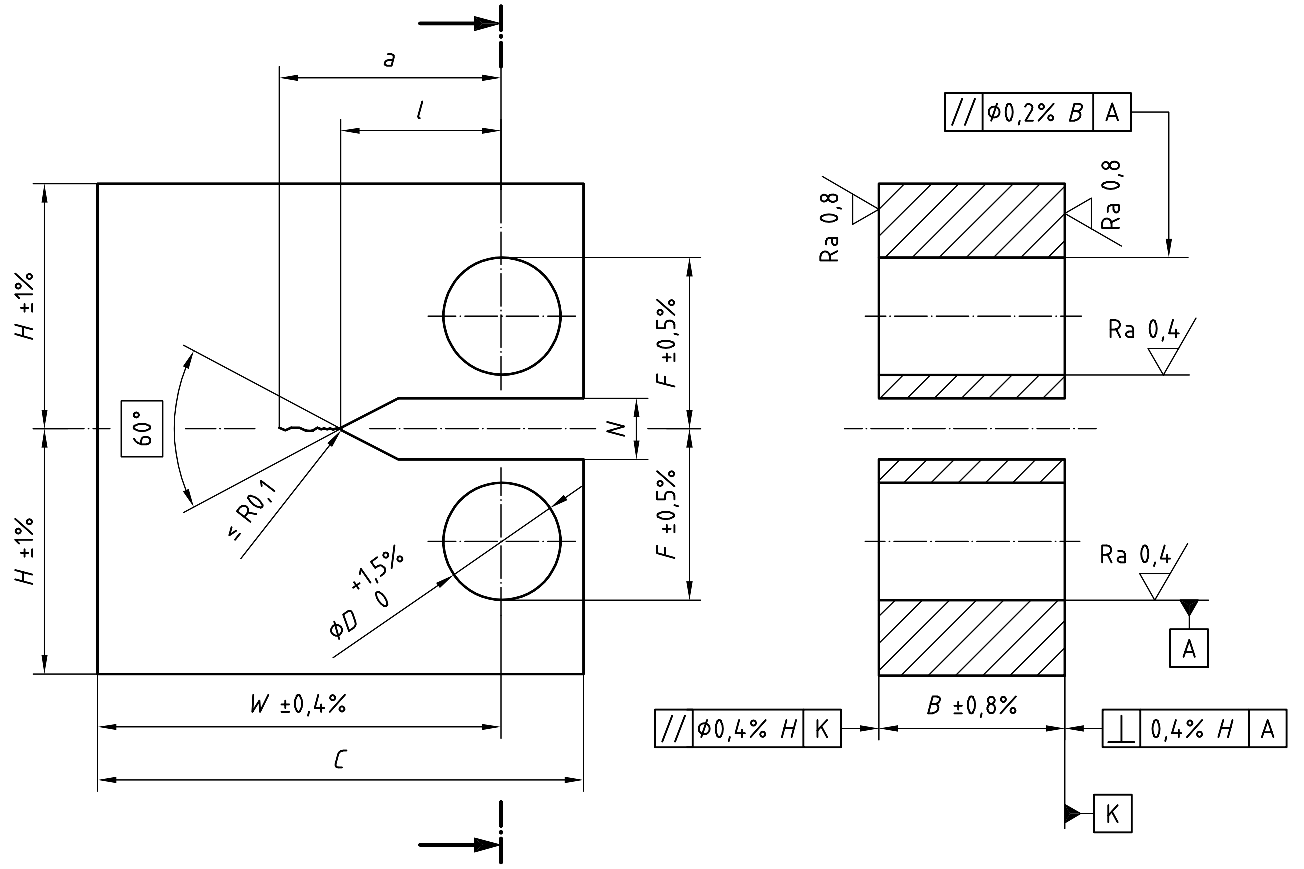
5.2.2.1 Specimen type

The test involves the use of a compact tension (CT) type specimen, according to the geometry given in Figure 4 (Figure 3 of ISO 7539-6:2003), with a W dimension of 26 mm and B equal to the greatest thickness allowed by the cylinder wall curvature and thickness, but not less than 85 % of the design thickness of the cylinder being qualified. The specimen orientation (Y-X) relative to the cylinder axis is shown in Figure 5.

At least 2 specimens diametrically opposed from the cylindrical wall shall be tested. Flattening of the specimen is not allowed. If the test specimen thickness cannot meet the validity requirements of ISO 7539-6, then the thickest possible specimen as specified above shall be tested.

Dimensions in millimetres

Surface roughness values in micrometres



Key

net width = W

total width, $C = 1{,}25\ W$ minimum

thickness, $B = 0{,}5\ W$

half height, $H = 0{,}6\ W$

hole diameter, $D = 0{,}25\ W$

half distance between hole outer edges, $F = 1{,}6\ D$

notch width, $N = 0{,}065\ W$ maximum

effective notch length, $l = 0{,}25\ W$ to $0{,}40\ W$

effective crack length, $a = 0{,}45\ W$ to $0{,}55\ W$

Figure 4 — Proportional dimensions and tolerances for compact tension test pieces

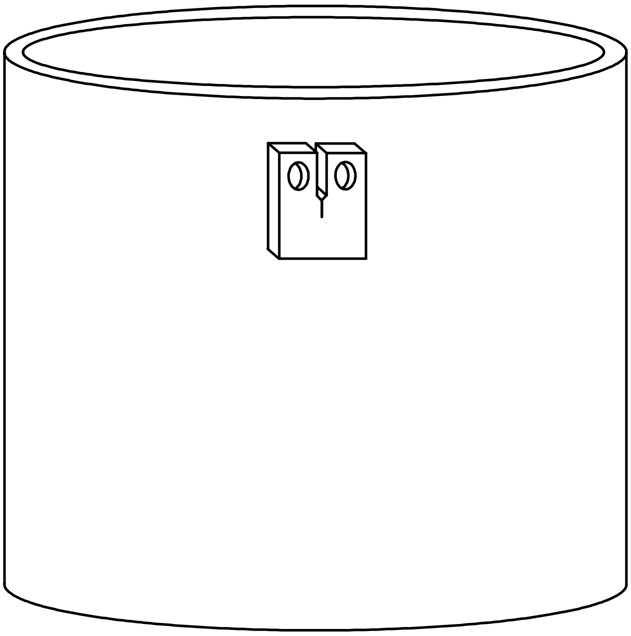


Figure 5 — Test piece: type and orientation

5.2.2.2 Specimen preparation

Prior to fatigue pre-cracking all necessary electrical contacts and wires required for crack monitoring shall be attached to the specimen. The specimen shall be thoroughly degreased, with particular attention paid to the notch tip region.

During all subsequent operations, especially the fatigue pre-crack, the specimen shall not be contaminated in order to ensure a clean crack front.

Fatigue pre-cracking shall be conducted in the test chamber in an atmosphere of hydrogen and meet the general requirements detailed in Clause 6 of ISO 7539-6:2003 for the initiation and propagation of fatigue cracks. The final maximum pre-cracking load shall give a final maximum stress intensity lower than the initial stress intensity required for the test.

On completion of fatigue pre-cracking, the specimen shall be measured as detailed in Clause 7 of ISO 7539-6:2003 to determine the thickness B, width W and average length of the fatigue pre-crack on the specimen surfaces. The last of these values shall be used in assessing the load necessary to produce the required initial stress intensity.

To prevent oxidation of the crack tip, the specimen shall now be put on test. If, for any reason, this is not possible, the specimen shall be stored in a clean, desiccated environment until ready for testing.

5.2.2.3 Gas purity and pressure

The hydrogen pressure in the test chamber shall be 150 bar, with a hydrogen purity of at least 99,999 5 %, and $O_2 \leqslant 1$ µl/l and $H_2O \leqslant 3$ µl/l. This gas shall be covered by a batch certificate of conformity.

As an alternative, gas with a 99,999 9 % purity ($O_2 \leqslant 0{,}1$ µl/l and $H_2O \leqslant 0{,}5$ µl/l) and covered by an individual certificate of conformity may be used.

5.2.2.4 Test apparatus

The tests shall be conducted in a stainless steel chamber, shown together with the loading bars and specimen in Figure 6, and installed on a servo-hydraulic test machine (Figure 7).

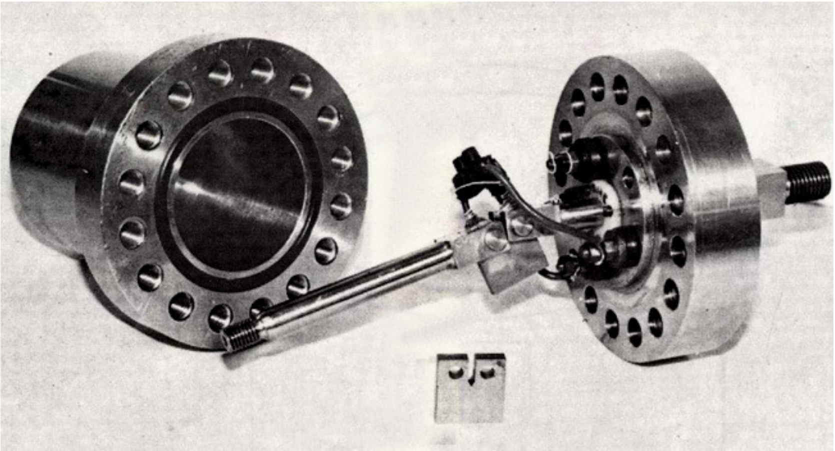


Figure 6 — Stainless steel chamber, showing loading bars and specimen

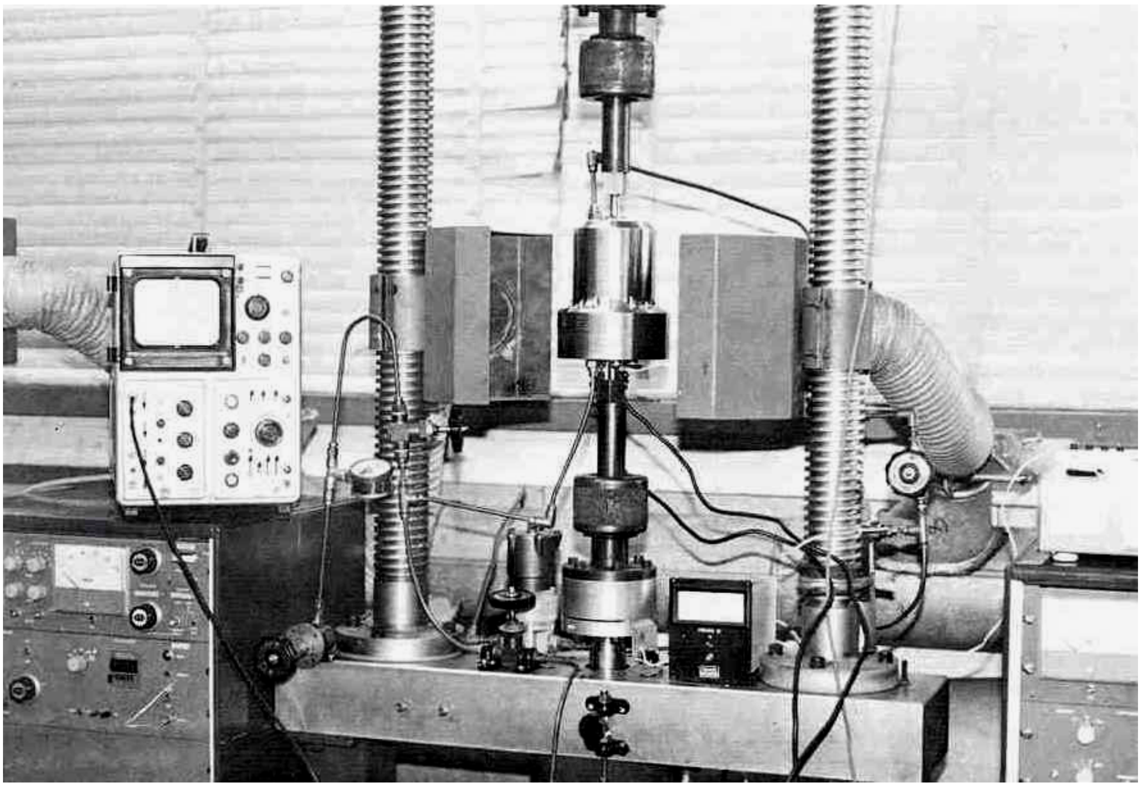


Figure 7 — Servo-hydraulic test machine

After installation of the specimen, the test chamber shall be evacuated to a pressure of < 130 Pa prior to the introduction of gaseous hydrogen. The purity of the gaseous environment in the test chamber shall be as specified in 5.2.2.3.

Crack initiation, and any subsequent growth, shall be detected and monitored using a direct current potential drop (DCPD) method.

5.2.2.5 Test method

If there is no prior knowledge of the material's threshold stress intensity factor, then the test shall be started using a load which produces a stress intensity factor of 1 MPa·m^{0,5} at the crack tip. Otherwise, the test may be started at a stress intensity factor equivalent to, but not exceeding, 50 % of the stress intensity factor derived from previous work.

The applied load shall be recorded continuously. The specimen shall be held at the initial load for a period of at least 20 min to allow for crack growth at room temperature. If no crack growth is detected by the end of the hold period, as evidenced by a plateau in the potential drop curve, the load shall be increased at a controlled rate in a step equivalent to a crack tip stress intensity factor increase of 1 MPa·m^{0,5}, and held for a further 20 min. This procedure shall be continued until failure occurs, with the appropriate load being recorded on the test data sheet.

The load shall be increased at the end of a hold period, by means of a servo-controller, at a constant rate of 2×10^{-3} kN·s^{-1}, until attainment of the crack tip stress intensity required for the subsequent hold period. This would result in a controlled crack tip stress intensity factor increase totalling 1 MPa·m^{0,5} over a time interval of approximately 1 min, assuming a test piece of thickness 7 mm and with an a/W of 0,5.

5.2.2.6 Specimen measurement

The specimen shall again be inspected in accordance with ISO 7539-6, with the appropriate measurements to 0,01 mm being made using a travelling microscope.

5.2.3 Test results

5.2.3.1 Analysis and acceptance criteria

Calculation of the test result shall be as stated in ISO 7539-6, through a relationship of the form

$$K_{1\mathrm{H}} = YP/BW^{0,5}$$

The load P used in the calculation is that applied to the specimen before the final increment that caused failure.

Provided the $K_{1\mathrm{H}}$ values of both CT specimens are greater than or equal to (60/950) × R_m (MPa·m^{0,5}), the material is qualified for a maximum tensile strength (R_m) equal to the average of the two tensile strength values obtained in 5.4. If the constraint validity requirement of 5.2.2.1 is not met, then the material is suitable up to the design thickness of the cylinder tested.

5.2.3.2 Test report

The detailed test conditions [gas purity and pressure, record of the direct current potential drop (DCPD) measurements, location and dimension of the specimen, $K_{1\mathrm{H}}$ and R_m (see 5.4) and the cylinder design drawing], including the material properties and chemical composition and the heat treatment conditions of the tested cylinder, shall be reported.

5.3 Test method to determine the resistance to hydrogen assisted cracking of steel cylinders (method C)

5.3.1 General

A fatigue-precracked specimen is loaded by a constant load or constant displacement method to a stress-intensity K_IAPP equal to a defined value. The specimen is kept in the loaded condition for a specified time in pressurized hydrogen gas at room temperature. After the test period, the specimen is examined to assess whether the initial fatigue crack did or did not grow.

If the test specimen exhibits less than or equal to a specified amount of crack growth, then the material is characterized as suitable for gas cylinders with respect to the hydrogen assisted cracking (HAC) resistance requirement.

Testing shall be conducted using applicable rules of ISO 7539-6 and additional rules specified in this document. Rules provided in ISO 7539-6 on corrosive environment need not be satisfied. Some symbols used in this method are to be found in Clause 3 of ISO 7539-6:2003.

5.3.2 Specimen configurations and numbers of tests

5.3.2.1 Fracture specimens

5.3.2.1.1 The compact tension (CT) specimen geometry given in Figure 4 (Figure 3 of ISO 7539-6:2003) shall be used. The specimen thickness shall not be less than 85 % of the design thickness of the cylinder which is being qualified.

5.3.2.1.2 Specimen orientation shall be Y-X in Figure 15 of ISO 7539-6:2003, for rectangular section.

5.3.2.1.3 At least three specimens taken 120° apart from the cylindrical wall shall be tested. Flattening of specimen blanks is not allowed.

5.3.2.1.4 If test specimen thickness cannot be obtained from the specified location or locations to meet the validity requirements of 5.3.4.6, then the thickest possible specimen shall be tested.

5.3.2.2 Tensile specimens

Three longitudinal tensile specimens taken adjacent to the CT specimens from the cylindrical wall area shall be used according to ISO 9809-1.

5.3.3 Fatigue precracking

All rules provided in Clause 6 of ISO 7539-6:2003 shall be met.

5.3.4 Specimen testing procedure

5.3.4.1 All rules in Clause 7 of ISO 7539-6:2003 shall be satisfied, except for those in 7.2.2, 7.2.6, 7.5.1, 7.5.2, 7.5.4, and 7.5.5.

5.3.4.2 Load the fatigue-precracked specimens to a stress-intensity K_{IAPP} determined from the following equation:

$$K_{\mathrm{IAPP}} = (60)^*(R_{\mathrm{m}}/950)$$

Specimens may be loaded by a suitable constant displacement or constant load method.

5.3.4.3 For specimens loaded by constant displacement method, the loading shall be determined by the following method.

a) At the end of the test, record the crack mouth opening displacement (CMOD) before unloading.

b) Unload the specimen.

c) Reload the specimen up to the measured CMOD in a device suitable for load measurement. Record the load and use this load in the K_{IAPP} calculations. This calculated K_{IAPP} shall be equal to or greater than the calculated K_{IAPP} value from 5.3.4.2.

5.3.4.4 Constant displacement loading

For testing of CTS specimens at a constant displacement loading, use the following equations:

$$V = \frac{\left(K_{\mathrm{IAPP}}\right)\sqrt{w}}{E\left[f(x)\right]\left(\sqrt{B/B_{\mathrm{N}}}\right)}$$

where

$$f(x) = (1 - x)^{0,5} (0{,}748 - 2{,}176\,x + 3{,}56\,x^2 - 2{,}55\,x^3 + 0{,}62\,x^4)$$

$$x = \frac{a}{w}$$

5.3.4.5 Constant loading condition

The stress-intensity factor equations provided in ISO 7539-6 shall be used in testing of CT specimens in the constant load test method.

5.3.4.6 All specimens shall meet the validity requirements specified in 7.6.6 e) of ISO 7539-6:2003, except as exempted in 5.3.4.1 above.

5.3.5 Test procedure

5.3.5.1 Place the test specimens in a high-pressure test chamber.

NOTE Extended periods of time after loading and before exposure to hydrogen gas may affect the final results.

Evacuate the test chamber to eliminate any traces of air or moisture absorbed by the walls. Scavenging with the test gas followed by vacuum pumping can be used to improve the cleaning efficiency.

Pressurize the test chamber with hydrogen gas to at least 150 bar. The O_2 content in the hydrogen gas shall not exceed 1 µl/l and H_2O content shall not exceed 3 µl/l.

5.3.5.2 The specimens shall be tested for at least 1 000 h at room temperature.

5.3.6 Crack growth examination

5.3.6.1 After the specified test period, unload the specimen and mark the HAC advance using one of the following methods.

a) Heat tinting the specimen at about 300 °C for 30 min.

b) Fatigue cycling at maximum stress-intensity factor not exceeding 0,6 K_{IAPP}. Advance the crack by at least 1 mm.

c) Fracturing the specimen at very low temperature such as that of liquid nitrogen.

5.3.6.2 Measure the crack growth, using a scanning electron microscope. Measurements shall be taken at locations perpendicular to the pre-crack at 25 %, 50 % and 75 % of B. Calculate the average of these three values.

5.3.6.3 If the average measured crack growth does not exceed 0,25 mm**,** the specimen passes the test.

5.3.7 Cylinder material qualification

5.3.7.1 If all specimens pass, the material is qualified for maximum tensile strength equal to the average of the three tensile strength values obtained in 5.3.2.2.

5.3.7.2 If the constraint validity requirement of 5.3.4.6 is not met, then the material is suitable up to the design thickness of the cylinder wall.

5.3.8 Test report

The information described in ISO 7539-6:2003, Clause 8, with the exception of 8 e), shall be reported. The report shall indicate if the validity criteria are met or not and shall include scanning electron microscope micrographs. The report must be kept on file permanently as a record that the cylinder material has been tested and found acceptable.

5.4 Tensile tests

Two longitudinal tensile specimens taken adjacent to the CT specimens (if applicable) from the cylindrical wall area shall be tested in accordance with ISO 9809-1.

(*Continued from second cover*)

International Standard	*Title*
ISO 11120 : 1999	Gas cylinders — Refillable seamless steel tubes for compressed gas transport, of water capacity between 150 l and 3 000 l — Design construction and testing

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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